NAREL Standard Operating Procedure
for Sodium Hydroxide Fusion of Solid Matrices
Prior to Actinide Analysis

AMS/SOP-20

Revision 2

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Revision History

Rev.	DCN	Coordinator	Date
0	AMS/SOP-20	Shane Knockemus	2016-06-16
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Changes Between Revisions 1 and 2	
Section	**Description of Changes**
8.11	Changed from: "Titanium (III) chloride, $TiCl_3$, (10–15 % in hydrochloric acid) [CAS# 7705-07-9]. Replace within six months after opening. Store in amber bottle away from direct sunlight." To read: "Titanium (III) chloride, $TiCl_3$, (20 % in hydrochloric acid) [CAS# 7705-07-9]. Replace within six months after opening. Store in amber bottle away from direct sunlight."
12.1.4	Deleted step: "Using tongs, carefully place crucibles (with lids) in furnace preheated to 600 °C."
12.2.6	Changed from: "Pipet 7 mL of 10 % $TiCl_3$ to each sample. Other strengths of $TiCl_3$ can be used if the amount of $TiCl_3$ is adjusted appropriately." To read: "Pipet 4 mL of 20 % $TiCl_3$ to each sample. Other strengths of $TiCl_3$ can be used if the amount of $TiCl_3$ is adjusted appropriately."
12.2.11	Removed last sentence: "This is a good place to stop if needed."
12.2.13	Changed from: "Pipet the following into each centrifuge tube: 0.5 mL of Ca^{+2}, 1 mL of La^{+3} carrier, and 3 mL of 10 % $TiCl_3$." To read: "Pipet the following into each centrifuge tube: 0.5 mL of Ca^{+2}, 1 mL of La^{+3} carrier, and 2 mL of 20 % $TiCl_3$."

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1.0 PURPOSE

1.1 This standard operating procedure (SOP) describes a sodium hydroxide fusion technique applicable to solid samples to be analyzed for isotopic uranium, plutonium, thorium, and/or americium.

2.0 SCOPE AND APPLICATION

2.1 The method applies to the sodium hydroxide fusion of solid samples prior to the chemical separation of the actinides.

2.2 The method is rapid and vigorous. It provides a total dissolution of sample. It is capable of digesting refractory particles that may be present in the sample.

2.3 This method provides total dissolution of a variety of matrices (soil, sludge, sediment, air filters, vegetation, tissue, and other solid environmental matrices).

3.0 DEFINITIONS

3.1 **R value** – the ratio of observed activity divided by the actual amount of added activity, a measure of recovery

NOTE: See *NAREL Common Terminology* (DR/T-1) for the definitions of other terms and acronyms used in this document.

4.0 SUMMARY OF METHOD

4.1 The method is based on the fusion of a homogenized aliquot of solid sample using sodium hydroxide fusion at 600 °C.

4.2 This method is for the fusion of a sample size of 1–1.5 g. Dissolution of larger aliquots can be achieved, but the amounts of reagents used will need to be adjusted accordingly.

4.3 Am, Pu, Th, and U are separated from the fused matrix by using an iron/titanium hydroxide precipitation followed by a lanthanum fluoride removal step.

4.4 The resulting solutions are then analyzed for Am, Pu, Th, and/or U depending on project requests.

5.0 INTERFERENCES

5.1 Because of unknown variability within sample groups and matrices there may be instances when slight deviations from this SOP may be advantageous in optimizing sample dissolution. Such instances may include (but are not limited to) varying the amounts of Fe^{+3}, La^{+3}, HNO_3-boric acid solution, Al^{+3}, or increasing the volume of the load solution with 3 M HNO_3. Any such instances must be noted in the appropriate logbook.

5.2 Americium and uranium analysis should not be analyzed from the same aliquot.

5.3 It is imperative the zirconium crucibles are properly cleaned before being used again. Improperly cleaned crucibles will lead to the contamination of future samples.

6.0 ROLES AND RESPONSIBILITIES

6.1 Unless otherwise noted, the radiochemist is responsible for performing all steps of this procedure. These responsibilities include grouping samples into QC batches, performing chemical separations and recording all data in laboratory notebooks.

7.0 EQUIPMENT AND SUPPLIES

7.1 Adjustable temperature laboratory hotplates.

7.2 Balance, top loading or analytical, readout display of at least ±0.01 g.

7.3 Centrifuge able to accommodate 50 and 250 mL tubes.

7.4 Centrifuge tubes, 50 mL and 250 mL capacity.

7.5 Zirconium crucibles with lids. Low form, 250 mL capacity. Other sizes are allowed.

7.6 100 µL, 200 µL, 500 µL, and 1 mL pipets or equivalent and appropriate plastic tips.

7.7 1–10 mL electronic/manual pipet(s).

7.8 Ice water bath or dry bath equivalent.

7.9 Muffle furnace capable of reaching at least 600 °C.

7.10 Tongs for handling crucibles (small and long tongs).

7.11 Tweezers or forceps.

7.12 Vortex stirrer.

8.0 REAGENTS AND STANDARDS

8.1 Reagent grade chemicals (or better) shall be used in all tests.

8.2 Reagent water in this method is laboratory de-ionized water which is treated by a point of use filtration system to ≥18.0 MΩ·cm resistivity (see the *De-ionized Water System* section in the *Equipment* chapter of the *NAREL Radiochemistry Quality Assurance Manual*) which is equivalent to ASTM Type 1, and shall be interference free. Analysis of a method blank must verify that the water is free from interferences.

8.3 Aluminum nitrate nonahydrate, $Al(NO_3)_3 \cdot 9H_2O$, [CAS# 7784-27-2].

8.3.1 Aluminum nitrate, (2 M): Dissolve 750 g of $Al(NO_3)_3 \cdot 9H_2O$ in ~600 mL of deionized water. Dilute to 1 L with deionized water.

8.4 Boric acid, H_3BO_3, [CAS# 10043-35-3].

8.5 Calcium nitrate tetrahydrate, $Ca(NO_3)_2 \cdot 4H_2O$, [CAS# 13477-34-4].

8.5.1 Calcium nitrate, (1.25 M): Dissolve 73.8 g of $Ca(NO_3)_2 \cdot 4H_2O$ in 100 mL of water and dilute to 250 mL with deionized water.

8.6 Hydrochloric acid, HCl, (concentrated, 12 M), [CAS# 7647-01-0].

8.6.1 Hydrochloric acid (3 M): Add 250 mL of concentrated HCl to 500 mL of deionized water and dilute to 1 L with deionized water.

8.6.2 Hydrochloric acid (1.5 M): Add 125 mL of concentrated HCl to 700 mL of deionized water and dilute to 1 L with deionized water.

8.7 Hydrofluoric acid, HF, (concentrated, 29 M), [CAS# 7664-39-3].

8.8 Lanthanum nitrate solution. 1000 µg/mL of La^{+3}.

8.9 Iron (III) nitrate nonahydrate, $Fe(NO_3)_3 \cdot 9H_2O$, [CAS#7782-62-8].

8.9.1 Fe^{+3} (50 mg/mL): Dissolve 181 g of $Fe(NO_3)_3 \cdot 9H_20$ in 200 mL of deionized water. Dilute to 500 mL with deionized water.

8.10 Nitric acid, HNO_3, (concentrated, 16 m), [CAS# 7697-37-2].

8.10.1 Nitric acid (7 M): Add 438 of concentrated HNO_3 to 400 mL deionized water. Dilute to 1 L with deionized water.

8.10.2 Nitric acid (3 M): Add 188 mL of concentrated HNO_3 to 800 mL of deionized water and dilute to 1 L with deionized water.

8.10.3 Nitric acid (3 M) / boric acid (0.25 M): dissolve 15.4 g H_3BO_3 and 190 mL of concentrated HNO_3 to 500 mL of deionized water. Dilute to 1 L with deionized water.

8.11 Titanium (III) chloride, $TiCl_3$, (20 % in hydrochloric acid) [CAS# 7705-07-9]. Replace within six months after opening. Store in amber bottle away from direct sunlight.

8.12 Sodium hydroxide, pellets, [CAS# 1310-73-2].

9.0 SAFETY

9.1 All procedures performed at NAREL must be conducted following the requirements detailed in the *NAREL Chemical Hygiene Plan* (HS/M-2) and the *NAREL Radiation Safety Manual* (HS/M-1). Safety precautions associated with handling of chemical reagents, solutions, and all samples are the primary responsibility of the analyst. Any spills or accidents involving hazardous, corrosive, or toxic material must be immediately resolved.

9.2 All NAREL laboratory personnel are expected to use good laboratory practices. Most of the safety training is provided by the SHEM officer. The analyst is expected to comply with all directives given by the SHEM officer, and must take necessary precautions to prevent exposure or injury to both self and co-workers.

9.3 Unnecessary or prolonged exposure to laboratory chemicals should be avoided.

9.4 Aluminum nitrate nonahydrate, $Al(NO_3)_3 \cdot 9H_2O$, [CAS# 7784-27-2], is a strong oxidizer; contact with other material may cause fire. Harmful if swallowed or inhaled. Causes irritation to skin, eyes and respiratory tract. Inhalation may result in coughing and shortness of breath. Ingestion may cause gastroenteritis and abdominal pain. Avoid contact with skin and eyes by using appropriate protective clothing and equipment. Use only with adequate ventilation. Keep separate from combustible, organic, or any other readily oxidizable materials. Store in a tightly closed container.

9.5 Boric acid, H_3BO_3, [CAS# 10043-35-3]. May damage fertility. May damage the unborn child. Obtain special instructions before use. If concerned or exposed, get medical advice or attention.

9.6 Calcium nitrate tetrahydrate, $Ca(NO_3)_2 \cdot 4H_2O$, [CAS# 13477-34-4], is a strong oxidizer; contact with other material may cause fire. Causes irritation to skin, eyes and respiratory tract. Harmful if swallowed or inhaled. Inhalation may result in coughing and shortness of breath, ingestion may result in nausea, vomiting and diarrhea. Avoid contact with skin and eyes by using appropriate protective clothing and equipment. Use only with adequate ventilation. Keep separate from incompatibles, combustibles, organic or other readily oxidizable materials. Store in a tightly closed container.

9.7 Hydrochloric acid, HCl, [CAS# 7647-01-0], is harmful if swallowed, inhaled, or ingested. It can cause serious damage to eyes and skin. Ingestion can cause burns around the mouth, throat, and esophagus with irritation and pain. Hydrochloric acid causes chemical burns following contact with skin and eyes. Inhalation can cause toxic effects and may be fatal. Use hydrochloric acid only with adequate ventilation and appropriate protective clothing. Always release caps slowly to ensure slow dissipation of vapors. Store concentrated hydrochloric acid in the original container, securely sealed, in a cool, dry, well-

ventilated area, away from alkaline materials, galvanized steel, and zinc. Avoid strong bases. Do not discharge into sewer or waterways.

9.8 Hydrofluoric acid, HF, [CAS# 7664-39-3], is a highly reactive chemical. It must be stored in plastic containers, and away from light, heat, and strong bases. Hydrofluoric acid is highly destructive to tissue and may be fatal if inhaled, swallowed, or absorbed through the skin. Hydrofluoric acid should be used only by persons trained and familiar with appropriate safety precautions.

9.9 Iron (III) nitrate nonahydrate, $Fe(NO_3)_3 \cdot 9H_2O$, [CAS# 7782-61-8]. May intensify fire. Oxidizer. Causes skin irritation. Causes serious eye irritation. May cause respiratory irritation. Store away from clothing or combustible material.

9.10 Lanthanum (III) nitrate hexahydrate, $La(NO_3)_3 \cdot 6H_2O$, [CAS# 10277-43-7]. May intensify fire. Causes skin irritation. May cause respiratory irritation. Avoid breathing dust. Store away from clothing or combustible material.

9.11 Nitric acid, HNO_3, [CAS# 7697-37-2], is poisonous, reactive, and a strong oxidizer. Contact with other materials may cause fire. It can cause burns to body tissues and may be fatal if ingested or inhaled. Vapors are irritating to eyes and mucous membranes. Use only with adequate ventilation and proper protective clothing and gloves. Nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, and combustible organics. Store away from light and heat.

9.12 Sodium hydroxide, pellets, NaOH, [CAS# 1310-73-2]. May be corrosive to metals. Causes severe skin burns and eye damage. Do not breathe dust. Wear protective clothing. Wash hands after handling.

9.13 Titanium chloride, $TiCl_3$, [CAS# 7705-07-9], is a flammable solid and can cause severe eye and skin burns. Replace within six months after opening. Store in amber bottle away from direct sunlight.

9.14 Safety data sheets (SDSs) are available to all personnel involved in chemical analysis. It is the responsibility of each analyst to be familiar with chemicals used during an analysis.

9.15 Refer to the *NAREL Chemical Hygiene Plan* (HS/M-2) for verification of appropriate safety and health practices.

10.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

10.1 Soil samples can be shipped to the laboratory in either plastic or glass containers. No preservation is required.

10.2 Special handling such as refrigeration or freezing may be required for samples of other matrices such as animal tissue or vegetation.

10.3 Samples for actinides analysis do not require refrigeration during storage.

11.0 CALIBRATION AND STANDARDIZATION

11.1 All fixed volume pipets used must be calibrated and checked *in accordance with the NAREL Standard Operating Procedure for Calibration, Use, and Maintenance of Pipets* (SE/SOP-4).

11.2 All balances used must be calibrated and checked in accordance with the *NAREL Standard Operating Procedure for Calibration of Balances* (SE/SOP-1).

12.0 PROCEDURE

12.1 *Fusion*

12.1.1 Weigh out sample aliquot(s) of predetermined size into a zirconium crucible. To facilitate a matrix for control samples (LCS, RBK), add 0.5 mL of 1.25 M $Ca(NO_3)_2$ solution to empty zirconium crucible.

12.1.2 Add appropriate tracers and/or spikes. Place crucible on a hotplate and heat to dryness on medium heat.

12.1.3 Add 15 g NaOH pellets to each crucible.

12.1.4 Allow samples to fuse at 600 °C for a minimum of 15 min. If necessary, samples can remain in the furnace for several hours.

12.1.5 Remove hot crucibles from furnace very carefully using tongs. Place the crucibles in a hood, remove lids, and allow to cool.

12.1.6 Add 25–50 mL of deionized water to each crucible. Place crucible on hotplate to loosen and dissolve fusion cake.

12.2 *Preconcentration of actinides from hydroxide matrix*

12.2.1 For each sample to be analyzed prepare a labeled 250 mL centrifuge tube. Pipet 2.5 mL of Fe^{+3} carrier (50 mg/mL) and 3 mL of La^{+3} carrier (1000 µg/mL) into each tube.

12.2.2 Carefully transfer the dissolved fusion cake from step 12.1.7 into the appropriately labeled centrifuge tube.

12.2.3 Rinse the crucible several times with water. Combine each rinse with the original sample in centrifuge tube.

12.2.4 Add 5–10 mL of either 3 M HCl or 3 M HNO_3 (the type of acid used to rinse the crucible has no quantitative impact). Place crucible on hotplate to warm the acid. Combine the warm acid rinse with the original sample in centrifuge tube. Rinse the crucible with additional water. Combine the rinse with the sample.

12.2.5 Dilute the centrifuge tube containing the fused sample to ~175 mL with deionized water.

12.2.6 Pipet 4 mL of 20 % $TiCl_3$ to each sample. Other strengths of $TiCl_3$ can be used if the amount of $TiCl_3$ is adjusted appropriately.

12.2.7 Cap and shake/mix each centrifuge tube.

12.2.8 Cool centrifuge tubes in an ice-bath for ~10 min.

12.2.9 Centrifuge the centrifuge tubes containing the samples for 3–5 min at 3500 rpm. Time and speed of centrifuge can be adjusted if necessary.

12.2.10 Decant the supernate. Retain the precipitate.

12.2.11 To the precipitate remaining from step 12.2.10, dilute to ~75 mL of 1.5 M HCl. Cap and shake the centrifuge tube to dissolve or break up precipitate.

12.2.12 Dilute to sample in centrifuge tube to ~175 mL with deionized water. Cap and shake centrifuge tube.

12.2.13 Pipet the following into each centrifuge tube: 0.5 mL of Ca^{+2}, 1 mL of La^{+3} carrier, and 2 mL of 20 % $TiCl_3$.

12.2.14 Add 20 mL 29 M HF.

12.2.15 Cap and shake/mix each centrifuge tube. Place centrifuge tubes in ice bath for ~10 min.

12.2.16 Remove from ice bath. Centrifuge for 3–5 min at 3500 rpm.

12.2.17 Decant the supernate. Retain the precipitate.

12.2.18 Pipet 7 mL of 3 M HNO_3 / 0.25 M H_3BO_3 solution to each centrifuge tube.

12.2.19 Cap and shake/mix each sample to dissolve the precipitate.

12.2.20 Transfer the sample to a labeled 50 mL centrifuge tube.

12.2.21 To rinse the 250 mL centrifuge tubes, pipet 7 mL of 2 M $Al(NO_3)_3$ and 7 mL of 7 M HNO_3. Cap and shake/mix. Combine rinse from each sample to the appropriate 50 mL centrifuge tube. If the sample contains undissolved material, add 5 mL of 3 M HNO_3.

12.2.22 The sample is now ready for chemical separation of Am, Pu, Th, and/or U. Proceed to the appropriate method SOP.

12.3 *Cleaning of Zirconium Crucibles*

12.3.1 Add approximately 10 mL of 16 M HNO_3 to each crucible.

12.3.2 Place the crucible on a hotplate set at a medium to high temperature setting.

12.3.3 Allow time for the HNO_3 to get sufficiently heated. Carefully swirl the hot HNO_3 around the crucible. Try to allow the hot HNO_3 to contact as much of the inside surfaces of the crucible as possible.

12.3.4 Discard the hot HNO_3 into a waste bucket for neutralization. Repeat the hot HNO_3 wash at least once.

12.3.5 After the final hot HNO_3 wash, while holding the crucible upside down over the waste bucket, carefully rinse all surfaces of the crucible (inside and outside) with ~6 M HNO_3 from a squirt bottle. Make sure all surfaces of the crucible are rinsed adequately.

12.3.6 Carefully rinse both sides of the crucible lid with 16 M HNO_3.

12.3.7 Rinse the lid with ~6 M HNO_3 from a squirt bottle. Make sure all surfaces of the lid are rinsed adequately.

12.3.8 Soak the crucible and lid for at least 30 minutes in a sink filled with hot water and a radiation decontaminating detergent. If necessary, scrub all surfaces of the crucible and lid with a sponge. Do not scrub any inside surfaces with a course material that could possibly etch the crucible or lid.

12.3.9 Place the crucible and lid in a dishwasher and wash for at least 3 cycles. After the crucibles and lids are removed from the dishwasher, rinse them with deionized water.

12.3.10 If any crucible shows signs of excessive wear, becomes etched, or is compromised in any way, the crucible must be removed from use.

13.0 QUALITY CONTROL PROCEDURES

13.1 Reference standards used to provide tracers, spiking solutions, standards, or calibration sources must be obtained from the National Institute of Standards and Technology (NIST) or suppliers who participate in supplying NIST standards or NIST traceable radionuclides.

13.2 For each QC batch of up to 20 samples of the same matrix, the analyst must add the following quality control samples:

13.2.1 method blank.

13.2.2 laboratory control sample (LCS).

13.2.2.1 At least one analyte must be included in any LCS. For analytical methods that measure more than one analyte, it is not necessary to include every analyte in the LCS; however, each analyte that is included must be evaluated.

13.2.2.2 The activity of an analyte added to the LCS must be at least ten times the normal expected minimum detectable activity (MDA) for that analyte and should be comparable to sample activities when sample activities in the batch are expected to be higher than ten times the MDA. The spike level should be high enough to ensure that under expected measurement conditions, the relative standard counting uncertainty will not exceed 5 %.

13.2.3 replicate sample (duplicate).

13.3 Analysts are required to control chart results from blanks and laboratory control samples, and to observe the control charts for indicators of possible problems in the measurement system. LIMS software allows the analyst to input data points and to view and print the control charts.

13.4 See the *NAREL Radiochemistry Quality Assurance Manual* (QA/QAM-1) for acceptance criteria for QC samples, and equations for calculating values for quality indicators.

14.0 DATA ANALYSIS AND CALCULATIONS

14.1 Not applicable

15.0 DATA REVIEW

15.1 Refer to *NAREL SOP for Review of Radiochemistry Data* (DR/SOP-2) for general data-review instructions. Refer to Section 15 of *NAREL Standard Operating Procedure For Isotopic Determination of Plutonium, Thorium, and Uranium in Solid Matrices Following Sodium Hydroxide Fusion* (AM/SOP-41) for more specific data-review instructions for this procedure.

16.0 RECORDS MANAGEMENT

16.1 The following documents are generated during this procedure:

- Actinide logbook pages
- Reagent logbook pages

16.2 When the batch is complete, deliver original copies of all records and documents, including copies of relevant logbook pages, to the CERLS Quality Assurance Coordinator (QAO). The QAO reviews the records and forwards them to the Radiochemistry Data Coordinator (RDC) for filing or inclusion in client data packages.

17.0 METHOD PERFORMANCE

17.1 See Section 17 of *NAREL Standard Operating Procedure For Isotopic Determination of Plutonium, Thorium, and Uranium in Solid Matrices Following Sodium Hydroxide Fusion* (AM/SOP-41).

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA places pollution prevention as the management option of first choice.

18.2 Volumes of prepared reagents are made in the smallest amounts consistent with sample batch sizes to minimize having to discard unused reagents.

19.0 WASTE MANAGEMENT

19.1 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. It is the responsibility of each laboratory to assure adherence to EPA regulations. Specific information can be found in the *NAREL Chemical Hygiene Plan* (HS/M-2).

19.2 The waste stream generated from analyzing one sample for the previously described procedure is 15 g NaOH, 125 mg Fe^{+3}, 4 mg La^{+3}, 100 mg Ca^{+3}, 20 mL concentrated HF, 50 mL of 1.5 M HCl, 7 mL of 2 M $Al(NO_3)_3$, 7 mL of 7 M HNO_3, 7 mL of 3 M HNO_3, 7 mL of 0.25 M H_3BO_3.

19.2.1 Waste solutions are collected in a bucket, neutralized, and poured down the drain.

20.0 REFERENCES

20.1 "Rapid Method for Sodium Hydroxide Fusion of Concrete and Brick Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents." www.epa.gov/narel/Docs/rapid_methods

20.2 *NAREL Radiation Safety Manual* (HS/M-1).

20.3 *NAREL Chemical Hygiene Plan* (HS/M-2).

20.4 *NAREL Radiochemistry Quality Assurance Manual* (QA/QAM-1).

20.5 *NAREL Common Terminology* (DR/T-1).

20.6 *NAREL SOP for Review of Radiochemistry Data* (DR/SOP-2).

20.7 *NAREL Standard Operating Procedure For Isotopic Determination of Plutonium, Thorium, and Uranium in Solid Matrices Following Sodium Hydroxide Fusion* (AM/SOP-41).

20.8 *NAREL Standard Operating Procedure for Calibration, Use, and Maintenance of Pipets* (SE/SOP-4).

20.9 *NAREL Standard Operating Procedure for Maintenance and Use of Balances* (SE/SOP-1).

20.10 *NAREL Standard Operating Procedure for Calibration and Use of Alpha Spectrometers Using AlphaVision* (NC/SOP-8).

21.0 APPENDICES (TABLES, DIAGRAMS, AND FLOWCHARTS)

21.1 Process Flowchart

Appendix 21.1

Process Flowchart